

# **Interfacial Properties of Electron Beam Cured Composites**

*CRADA ORNL99-0544  
FY00 1<sup>st</sup> Quarter Progress Report*

*to*

*DOE Laboratory Technology Research Program*

*Air Force Research Laboratory*

*and*

*NASA Langley Research Center*

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## **1. Executive Summary**

The objectives of the CRADA are to:

- Confirm that fiber-resin adhesion is responsible for the observed poor shear properties;
- Determine the mechanism(s) responsible for poor adhesion between carbon fibers and epoxy resins after electron beam (EB) curing;
- Develop and evaluate resin systems and fiber treatments to improve the properties of EB cured, carbon-fiber-reinforced composites; and
- Develop refined methods for processing EB cured, carbon-fiber-reinforced composites.

The CRADA team has been organized into integrated project teams (IPT's) with each team focused on specific research tasks.

- The Adhesion IPT, led by Dr. Mark Wilenski (Boeing), is focused on understanding why there is characteristic poor adhesion between the fiber and resin in EB cured composites, and how to improve the adhesion.
- The Irradiation IPT, led by Vincent Lopata (Acsion), is focused on evaluating the effects of irradiation parameters on composite properties, especially interlaminar shear and toughness.
- The Materials & Processing (M&P) IPT, led by Christopher Janke (ORNL), is focused on developing constituent materials and processing methods that lead to better composite properties, especially resin toughness.
- The Leadership IPT, led by Cliff Eberle (ORNL) and Paige Kirn (Lockheed Martin), consists of the respective technical IPT leaders and project management staff. It is responsible for coordinating project activities among the three technical IPT's and ensuring that all activities are designed to achieve progress toward accomplishment of the essential project objectives.

The Adhesion IPT has made significant progress toward both understanding the cause of the lower mechanical properties of EB cured composites and developing methods for improving these properties. Progress has been made on understanding reaction poisoning, and processing effects, as well as evaluating carbon fiber surface conditions. Thermal analog materials are being prepared and tested to establish benchmark property values under realistic processing conditions (i.e., typical properties of competing materials). Multiple approaches for characterizing and re-engineering the fiber-matrix interface are underway, and the preliminary data is encouraging. Several specimens, that were prepared using various adhesion promoting schemes, have been tested and exhibited significantly improved mechanical properties. Industrial scale-up issues are also beginning to receive attention.

The Irradiation IPT provided irradiation support for various adhesion studies. Some of the irradiators completed test runs to validate conformity among the irradiators, as well as the software code that was reported previously. Additional data will be acquired in the Jan. – Mar. quarter. A paper on the irradiation experiments and software validation was submitted for presentation at the SAMPE 2000 symposium. Planning is underway for “round robin” irradiation and testing of materials at all irradiators’ facilities in the Jan. – Mar. quarter.

The Materials & Processing IPT has completed the formulation and EB curing of several toughened, EB curable cationic epoxy resins and neat resin  $K_{IC}$  fracture toughness testing is currently underway. A parametric study evaluating several laminate lay-up and EB cure variables and their effects on the void content, fiber-matrix adhesion, and transverse tension of 24 ply, unidirectional, IM7-GP-12K/3K resin laminates is in progress.

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The CRADA team is on track to timely achievement of scheduled milestones and is presently operating under budget. Discussions with the Army Research Laboratory (ARL), regarding its prospective entry into the CRADA as a new sponsor and team member, continued during this quarter. ARL is expected to join the CRADA team in April. The ARL contributions will be used to investigate curing mechanisms, kinetics, and their impacts on material properties.

## **2. Adhesion**

The Adhesion IPT has made significant progress toward both understanding the cause of the lower mechanical properties of EB cured composites, and developing methods for improving these properties. The recent progress toward these objectives is shown in the following sections.

### **2.1. Adhesion IPT Mission**

The mission of the Adhesion IPT is to improve the adhesion of EB cured graphite/epoxy composites so that they meet or exceed their thermally cured counterparts. The researchers will determine the source of the low adhesion in current EB cured systems, and will use this understanding to improve their adhesion. Improvement methods will be economical, environmentally friendly, applicable to multiple resin/fiber systems, and will be compatible with the beneficial aspects of EB curing.

### **2.2. Technical Strategy**

The technical strategy for improving fiber-resin adhesion is to determine the source of the low adhesion in current EB cured systems, and to use the new knowledge to improve fiber-resin adhesion, as follows.

- Interface characterization – The fiber-resin interface will be chemically and mechanically characterized in order to understand the mechanisms responsible for and the nature of the adhesion deficiency.
  - Baseline database – Adhesion data will be compiled for a selected thermally cured system and used as a baseline target.
  - Fiber surface chemistry – The extent of adsorption and deactivation of the carbon fiber surface chemistry under irradiation will be determined using tools such as XPS, DSC, and chemisorption studies. Thermal desorption, mass spectrometry, Raman microscope spectroscopy, and thermogravimetric analysis studies will be performed to characterize fiber surface contamination.
  - Fiber-resin adhesion – Fiber-resin adhesion will be characterized via single fiber fragmentation and single fiber compression tests.
  - Interfacial failure mechanism(s) – Interfacial failure mechanisms will be identified using ultramicrotomy of the single fiber coupons coupled with transmission electron microscopy (TEM) observation to determine the locus of failure. The researchers will hypothesize a predictive model based on the physical and chemical changes that occur in the interphase during irradiation and are responsible for the reduction in fiber-matrix adhesion.
- Interface modification – The fiber-resin interface will be re-engineered by one or more of the following techniques to resolve the adhesion deficiency.
  - Fiber surface treatments – Fiber surface treatments will be specifically designed for promoting adhesion to EB curable epoxy resins. Fiber surface treatments involve a chemical modification of the nascent fiber surface that alters the surface chemical composition, such as oxidation, acid-base reactions, and etching, without a deliberate coating of the surface.
  - Fiber coatings – Fiber coatings, perhaps including coupling agents, reactive finishes, and sizings, may be optimized for EB curing.

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- Resin – The resin parameters that are most likely to affect adhesion will be systematically investigated and adjusted. These include surface energy, viscosity, and chemical functionality of the base resins and additives.

### **2.3. Technical Accomplishments**

#### **2.3.1. Understanding the Problem**

##### **2.3.1.1. Reaction Poisoning Effects**

- Studies into the effect of nitrogen containing species on the cationic reaction are beginning. Existing data shows that some amine containing species "poison" the reaction at some concentrations, but only retard it at others. This work will help determine what chemistries can be used to solve the adhesion problem.

##### **2.3.1.2. Carbon Fiber Surface Conditions**

- Fibers have been irradiated in a bath containing acetone and the CD1012 initiator. The samples were then washed using soxhlet extraction and analyzed using XPS (X-ray Photoelectron Spectroscopy). Data analysis is underway.

##### **2.3.1.3. Processing Effects**

- The effect of ambient cure temperature on adhesion is being investigated. An effect may be seen due to either alterations in resin mechanical properties, or surface wetting.
- The effect of dose rate and total dose on adhesion has been investigated prior to this effort. This testing has been repeated and expanded. Tests for the level of adhesion and  $T_g$  have been performed.
- The effect of debulk cycles on interfacial adhesion will be determined by using specimens already fabricated by the M&P IPT. Identification of the appropriate samples for test is beginning.
- The effect of a thermal post-cure on the interfacial adhesion is being investigated. Existing specimens will be subjected to potential use temperatures to determine the stability of both the resin and adhesion bond.

##### **2.3.1.4. Quantification of Target Thermal Properties**

- Specimens of common thermally cured epoxy/graphite composites are being gathered and tested to provide target values for the adhesion improvement efforts. Specimens of 3501-6, 977-2, 977-3, and 8552 with AS4 fiber are being sought from the end-users and material suppliers.
- Neat resin specimens of the same thermal resin systems are being sought for comparison with the performance of EB cured resins.

#### **2.3.2. Problem Resolution**

Methods aimed at mitigating the various causes of low adhesion are being investigated concurrently with the research into the cause of the low adhesion. Currently these methods include the use of isocyanates, amines, and sol-gels to form a strong bond between the fiber and resin as well as the addition of surfactants and tougheners to the bulk resin.

- Twenty-seven specimens with sizings based on isocyanate/epoxy chemistries have been fabricated and tested. Significant improvements in adhesion strength have been shown for a few of the systems. These results are being repeated for confirmation.
- Specimens with 3 different sol-gel sizings have been fabricated and tested. Moderate, but significant levels of improvement have been seen. Validation of these results is being performed.

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- Specimens made with resins containing small amounts of 2 different surfactants have been fabricated and tested. Moderate, but significant levels of improvement have been seen. Validation of these results is being performed.
- Specimens with 2 simple tougheners added to the bulk resin have been fabricated and tested. The results are being analyzed to account for the changes in bulk resin mechanical properties and will be reported in the future.

Alternative methods for testing adhesion are being investigated to allow confirmation and scale-up of the ITS (microdebond) test currently being used.

- The cruciform test being developed at AFRL tests a single fiber with the interface in tension. The test is still being developed, but significant progress has been made.
- The use of fabric laminates for shear testing is being investigated. Fiber with a removable sizing has been identified and the weaving of fabric is being pursued. Once available, the sizing will be removed and laminate specimens will be fabricated for test.

### **2.3.3. Industrial Scale-Up Concerns**

Any method used to improve the interfacial adhesion in EB cured composites must be scaleable to industrial levels. Concerns about production rates, hazardous materials, process robustness, and cost must be addressed prior to implementation of any improvement method. A document is being created to identify the issues associated with scaling up the various improvement methods. Another document will contain the perceived performance risks associated with these methods. For instance, the use of certain chemical modifiers may reduce the hot/wet performance of the composite.

## **2.4. Conclusion**

The Adhesion IPT has generated a large amount of data and is well on the way to understanding both the problem and solution to the problem at hand. No programmatic issues are foreseen at this time, and it is anticipated that the April 2,000 milestone will be achieved during the Jan. – Mar. quarter. The CRADA Adhesion team is pulling together resources from a variety of sources and is strongly leveraging work from external efforts. This cooperative approach greatly benefits all involved parties and will be continued throughout the program.

## **3. Irradiation**

### **3.1. Irradiation IPT Mission**

The goals of the irradiation IPT are:

- to provide EB processing support for the CRADA;
- to determine the effect of EB processing parameters on material parameters; and
- to determine the EB processing parameters required to give the optimum composite performance properties.

### **3.2. Technical Strategy**

The technical strategy for irradiation is to determine the sensitivity of material properties to irradiation parameters, and to develop robust irradiation procedures for reliably producing materials with predictable and satisfactory properties. Parameters to be studied may include:

- Accelerator-controlled parameters, including total dose, mean dose rate, instantaneous dose rate, beam energy, electrons vs. x-rays, etc;
- Parameters in the materials, including dose profile, electric charge deposition, temperature, moisture, voids/gases, etc; and
- Environmental parameters such as temperature, pressure, moisture, and atmospheric composition.

### **3.3. Technical Accomplishments**

#### **3.3.1. Support Services**

Acision provided laboratory space and radiation services to Dr. Brigitte Defoort (Michigan State University) as part of the Adhesion IPT.

#### **3.3.2. Accelerator Conformity Experiments**

The main objectives of these experiments are to determine the effects of radiation processing conditions on the:

- degree of cure of a resin,
- effect on thermal performance of a resin, and
- temperature profile during the curing of a resin.

Secondary objectives include:

- determining the effects of radiation processing conditions on composite mechanical performance,
- determining the effects of curing at higher temperatures, and
- determining the effects of resin curing with and without fiber.

The experimental program will look at 2 resin formulations, Tactix 123/CD1012 (3 phr) and Tactix 742/OPPI (3 phr). The 2 resin systems represent the extremes in viscosities expected for typical resins. The polymerization reactions are diffusion-controlled, therefore the viscosity of the resins will have a significant role to play. The resins will be cured under the following conditions:

- Dose increment – 5 and 25 kGy per pass;
- Time between passes – AFAP, 2, 5 and 10 minutes;
- Temperature – 25°C.

Resin samples will be sent to each of the EB facilities and cured according to the aforementioned conditions. The resins will then be analyzed by dynamic mechanical analysis to determine the effects. If there is partial curing in the two resins, there will be a peak in the loss modulus curve at around 90°C. The higher the peak at this temperature signifies lowering polymerization of the resin. Where possible the temperature rise of the resins will be monitored during the curing process.

Acision Industries has done some preliminary work on these experiments. The results for the Tactix 123/CD1012 (3 phr) system were included in the Oct. – Dec. 1999 report. Their results on the Tactix 742/OPPI(3 phr) will be included in the next scheduled report.

### **3.3.3. Code Validation Experiments**

The objective of these experiments is to provide radiation processing information to validate the radiation prediction code. A series of experiments were carried out at each of the EB facilities to provide information of beam shape and accumulated doses at prescribed distances from the accelerator. The conditions were then entered into the radiation prediction code to determine if the code could accurately predict the doses.

Two partners, Acsion Industries and STERIS Isomedix, have provided information. The other 2 partners, E-BEAM Services and Boeing, will provide the information in Feb. 2000. The information provided showed that there was good correlation between the code and actual data for distances at 30 and 45 cm from the exit window for the Acsion accelerator. Near the scan horn exit, the predicted doses were significantly different. Work is still being conducted to modify the code.

### **3.3.4. SAMPE Paper**

The paper titled "A Method for Specifying Consistent Radiation for the Processing of Electron Beam Cured Composites" has been submitted to SAMPE for presentation at the Spring 2000 Symposium.

## **4. Materials & Processing**

The Materials & Processing IPT has completed the formulation and EB curing of several toughened, EB curable cationic epoxy resins and neat resin  $K_{IC}$  fracture toughness testing is currently underway. A parametric study evaluating several laminate lay-up and EB cure variables and their effects on the void content, fiber-matrix adhesion, and transverse tension of 24 ply, unidirectional, IM7-GP-12K/3K resin laminates is in progress.

### **4.1. Materials & Processing IPT Mission**

The goal of the M&P IPT is to develop improved EB curable epoxy resin systems and processing methods for producing EB cured composites which meet or exceed the thermomechanical properties of autoclave cured, carbon fiber reinforced composites containing toughened epoxy resins 977-2 or 977-3, or untoughened 3501-6 epoxy (interim target only). To achieve this goal particular emphasis will be placed on overcoming the toughness limitations of the current EB resins without impairing the balance of modulus, strength, thermal stability, and processibility.

### **4.2. Technical Strategy**

The technical strategy for materials and processing is to toughen the epoxy resin systems while maintaining balance of properties, and to refine materials processing procedures, as follows.

- Resin toughening will be attempted by changing the resin backbone via chain extension agents or co-reactants, and/or by the incorporation of various toughening materials.
- Proposed resin modifications for affecting adhesion, conducted within the adhesion IPT, will be reviewed for their effects on balance of properties.
- Materials processing steps including prepregging, materials handling, laminate fabrication, and laminate consolidation will be investigated to determine if present techniques are introducing detrimental effects, or if alternative techniques produce superior results.

### **4.3. Technical Accomplishments**

#### **4.3.1. Materials**

The CRADA Materials & Processing IPT has completed the formulation and EB curing of several toughened, EB curable cationic epoxy resins. These systems are awaiting neat resin  $K_{IC}$  fracture toughness testing using the standard ASTM test method D 5045-93. Several thermal cured control resins, which are well known in the aerospace industry, will be tested concurrently with these materials. These include Hexcel's untoughened 3501-6 and toughened 8552 and Cytec Fiberite's toughened 977-2 and 977-3 epoxies. Various toughening strategies have been incorporated into this first series of EB curable resins including the use of thermoplastic particulates, polymer chain extension, and interpenetrating polymer networks. Additional resin systems currently being formulated or being considered for evaluation include other polymer chain extended resins, rubber modified resins, and thermoplastic interleaved prepregs. The top candidates from these evaluations will be further optimized in terms of thermal and mechanical performance, then combined with carbon fiber for composite testing.

#### **4.3.2. Processing**

A parametric study evaluating several laminate lay-up and EB cure variables and their effects on the void content, fiber-matrix adhesion, and transverse tension of 24-ply, unidirectional, IM7-GP-12K/3K resin laminates is in progress. Table 1 details the various laminates that have been fabricated and EB cured. All 18 laminates were cured under vacuum to 150 kGy at Acsion Industries. The parameters that were considered in this study included: lay-up operator; intermediate vacuum debulk intervals, times, and temperatures; additional intermediate debulk conditions; final vacuum debulk times and temperatures; additional pressure conditions; resin bleed and nonbleed conditions; lay-up and debulk locations; and EB cure dose per pass (this parameter influences the laminate temperature). Specimens from this activity are currently being tested for percent void content via acid digestion and mercury intrusion at YLA, Inc. and Acsion Industries, respectively. Additional specimens are also being tested by MSU to determine their level of fiber-matrix adhesion, using the ITS test, and by the U.S. Air Force to determine transverse tension strength.

## **5. Project Management**

The CRADA project management team exists to serve the technical team and ensure that the various technical activities are coordinated/integrated and are executed with proper fiscal and temporal discipline. The project team is expected to demonstrate satisfactory progress toward top-level milestones.

### **5.1. Status of Milestones and Deliverables**

Progress toward achieving the project objectives is measured monthly against the milestones and deliverables described in Table 2. The milestones and deliverables are themselves reviewed, on a semi-annual basis, for their appropriateness as indicators of progress toward achieving the project objectives. The first milestone due is the completion of fiber-resin interface chemistry studies on incumbent<sup>1</sup> materials, due in April of 2000. Current projections are that this milestone will be met ahead of schedule.

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<sup>1</sup> "Incumbent" materials are materials that already existed upon commencement of the project.

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**Table 1. Layup and EB Cure Parametric Study of Unidirectional, 24-ply IM7-GP-12K/3K Laminates (18)**

Panel #	Layup Operator	Interm. vacuum debulk interval (# of plies)	Interm. vacuum debulk time (min.)	Interm. vacuum debulk temp. (C)	Additional interm. debulk conditions	Final vacuum debulk time (min.)	Final vacuum debulk temp. (C)	Addl. pressure applied during final debulk @ 70C (160F) (psi)	Bleed vs. nonbleed during final debulk	Layup & debulk location	Cure dose per pass (kGy)	Panel temp. after 1st 25 (or 50) kGy and max. temp. (C)
1	BJF	4	15	RT	NA	15	RT	0	nonbleed	ORNL	25	55, 80
1	CJJ	4	15	RT	NA	15	RT	0	nonbleed	ORNL	25	55, 80
2	BJF	2	15	RT	NA	60	70	0	bleed	ORNL	25	55, 80
2	CJJ	2	15	RT	NA	60	70	0	bleed	ORNL	25	55, 80
3a1	BJF	4	15	RT	NA	60	70	0	bleed	ORNL	25	55, 80
3a1	CJJ	4	15	RT	NA	60	70	0	bleed	ORNL	25	55, 80
3a2	BJF	4	15	RT	NA	60	70	0	bleed	ORNL (2nd hot debulk done at Acision w/o bleed, cooled before cure)	25	55, 80
3a3	BJF	4	15	RT	NA	60	70	0	nonbleed	ORNL	25	55, 80
3b	BJF	4	15	RT	NA	60	70	0	bleed	ORNL	5	55, 67
3c	BJF	4	15	RT	NA	60	70	0	bleed	ORNL	50	90, 135
4a	BJF	1	15	RT	NA	60	70	0	bleed	ORNL	25	55, 80
4a	CJJ	1	15	RT	NA	60	70	0	bleed	ORNL	25	55, 80
4b	BJF	1	5	RT	NA	60	70	0	bleed	ORNL	25	55, 80
5	BJF	1	15	RT	every 4 plies at 70C for 60 min. w/ bleed	60	70	0	bleed	ORNL	25	55, 80
6	BJF	4 w/ bleed	60	70	NA	60	70	0	bleed	ORNL	25	55, 80
7	BJF	4	15	RT	NA	60	70	100 (press pressure applied until cooled to 32C)	nonbleed	ORNL	5	55, 67
8	BJF	24	15	RT	NA	60	70	0	bleed	ORNL	25	55, 80
9	BJF	4	15	RT	NA	60	70	0	bleed	Acision	25	55, 80

Panels were 6" x 6" (#1-12" x12"); 4 bleeders used per side (# 5 and 6 - 1 bleeder/side on 1st debulk, then 1 bleeder only on top side for others); used matls./methods specified in 1st EB CRADA; all panels were cured under vacuum to 150 kGy at Acision

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**Table 2. Project Milestones and Deliverables**

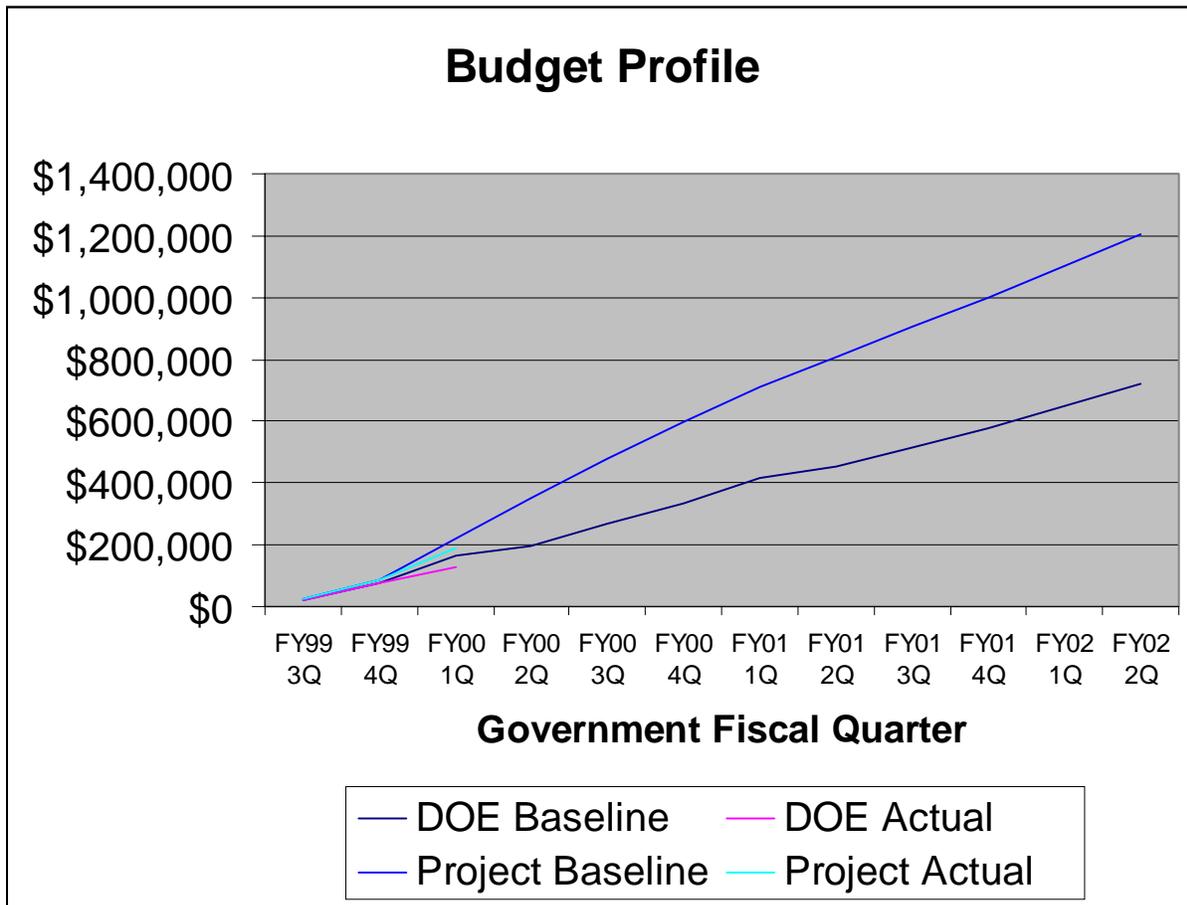
<i>Milestone</i>	<i>Metric</i>	<i>Deliverable</i>	<i>Date</i>
Fiber-resin interface chemistry studies on incumbent materials complete	Adsorption, deactivation of carbon fiber surface chemistry at specified processing conditions quantified & documented	Milestone report including data, analysis, conclusions, recommendations	4-19-00
Interface characterization of incumbent materials complete	Characterization matrix fully populated, <b>OR</b> source of ILSS deficiency understood	Milestone report including data, analysis, conclusions, recommendations	10-19-00
Incumbent materials processing development complete	Processing matrix fully populated with incumbent materials data	Milestone report including data & processing methods, analysis, conclusions, recommendations	10-19-00
Fiber surface treatments, chemical agents, and/or resins developed	Coupon data indicating ILSS increased $\geq 15\%$ compared to baseline*	Milestone report including data, analysis, conclusions; IP documented & protected	4-19-01
Composite system optimization complete	Coupon data indicating ILSS increased $\geq 20\%$ compared to baseline* <b>OR</b> composite optimization matrix fully populated	Final report including data, analysis, conclusions, recommendations; IP documented & protected	4-19-02
Processing development complete	Coupon data indicating ILSS increased $\geq 25\%$ compared to baseline* <b>OR</b> processing matrix fully populated with new materials data	Final report including data, analysis, conclusions, recommendations; IP documented & protected	4-19-02

\* Baseline is best EB processed material properties demonstrated with incumbent materials (same base resin and fiber) before new materials or processes are developed.

### **5.2. Budget Tracking**

Figure 1 below indicates the budget status for funds accountable to and expended by ORNL. The upper set of lines shows the budget baseline and spending profile for funds from all sources, including the DOE, other government agency sponsors, and funds-in from industrial partners. The upper set of lines shows the budget baseline and spending profile for DOE funds.

The project presently is about \$34,000 under budget. Approximately \$12,000 of this is attributable to delayed subcontract billing.



**Figure 1. Budget Baseline**

**5.3. Accomplishments**

- Conducted semi-annual CRADA meeting, Oct. 25, 1999, hosted by STERIS Isomedix, Chicago.
- Coordinated CRADA activities via weekly teleconferences; tracked technical progress and status of action items.
- Continued discussions with ARL regarding their stated desire to join the CRADA team.

**5.4. Issues**

- No significant new issues have arisen.

**5.5. Scheduled Events**

- Paper to be presented at the SAMPE 2000 Symposium, May 25, Long Beach, CA
- Semi-annual CRADA meeting, May 26, hosted by Applied Poleramic and YLA, Benicia, CA.
- Electron Beam Curing of Composites Workshop, June 13 – 15, Oak Ridge, TN

## **Appendix A**

### List of Acronyms

<b>AFRL</b>	<i>Air Force Research Laboratory</i>
<b>ARL</b>	<i>Army Research Laboratory</i>
<b>ASTM</b>	<i>American Society for Testing and Materials</i>
<b>CRADA</b>	<i>Cooperative Research and Development Agreement</i>
<b>DOE</b>	<i>Department of Energy</i>
<b>DSC</b>	<i>Dynamic Scanning Calorimetry</i>
<b>EB</b>	<i>Electron-Beam</i>
<b>ILSS</b>	<i>Interlaminar Shear Strength</i>
<b>IP</b>	<i>Intellectual Property</i>
<b>IPT</b>	<i>Integrated Project Teams</i>
<b>ITS</b>	<i>Interfacial Test System (microindentation method)</i>
<b>M&amp;P</b>	<i>Materials &amp; Processing</i>
<b>ORNL</b>	<i>Oak Ridge National Laboratory</i>
<b>SAMPE</b>	<i>Society for the Advancement of Material and Process Engineering</i>
<b>TEM</b>	<i>Transmission Electron Microscopy</i>
<b>XPS</b>	<i>X-ray Photoelectron Spectroscopy</i>

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**Appendix B**

CRADA Team

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*Interfacial Properties of Electron Beam Cured Composites*

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